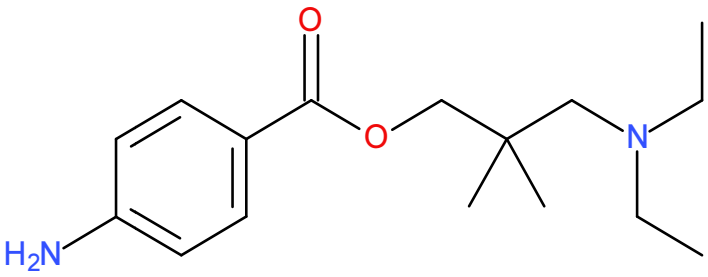
Dimethocaine



3-(diethylamino)-2,2-dimethylpropyl 4-aminobenzoate

Formula: $C_{16}H_{26}N_2O_2$
Formula weight: 278.39
Chemical Abstracts No.: 94-15-5 (base)
553-63-9 (* HCl)
Smiles code: Nc1ccc(cc1)C(=O)OCC(C)(C)CN(CC)CC
InChi key: OWQIUQKMMPDHQQ-UHFFFAOYSA-N
Other names: Larocaine, DMC

Two samples were investigated.

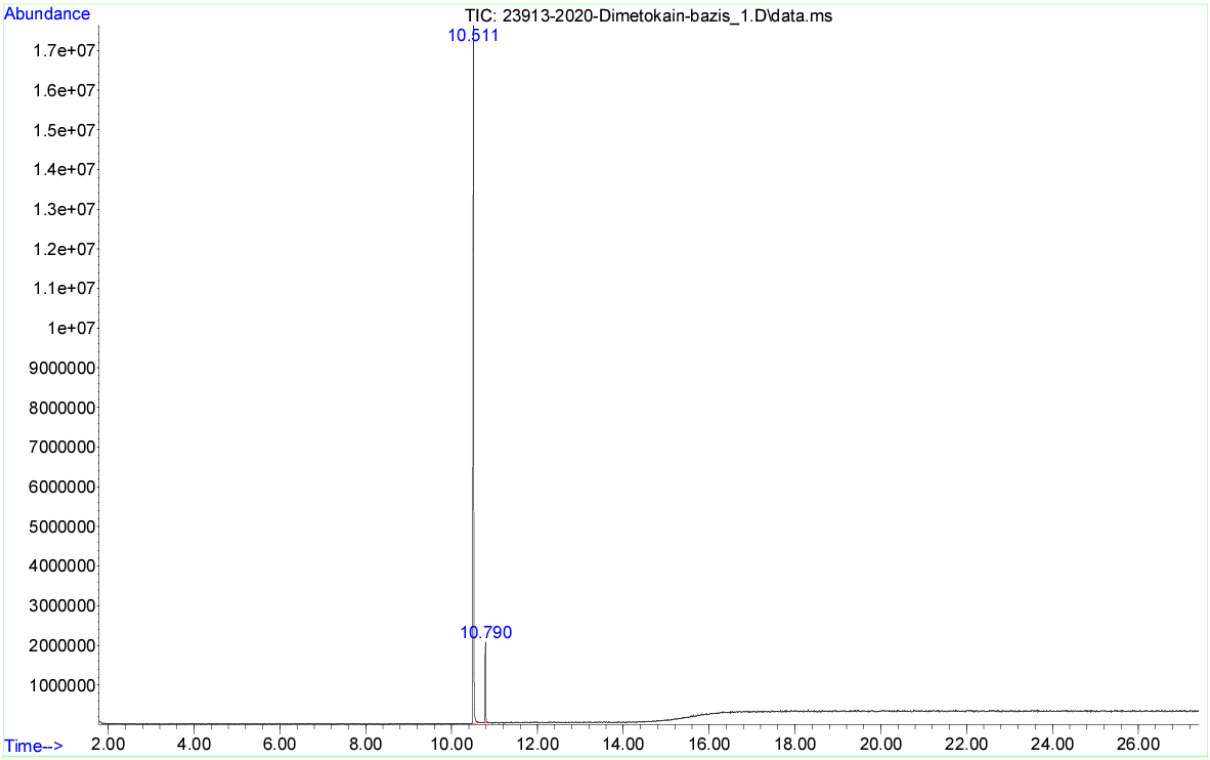
The evidence was 10.19 grams yellow chrystalline powder seized by Hungarian Tax and Customs at Budapest Liszt Ferenc Airport from postal matter.

Our reference standard was the LGC 2011 standard sample in HCl salt form.

GC-MS

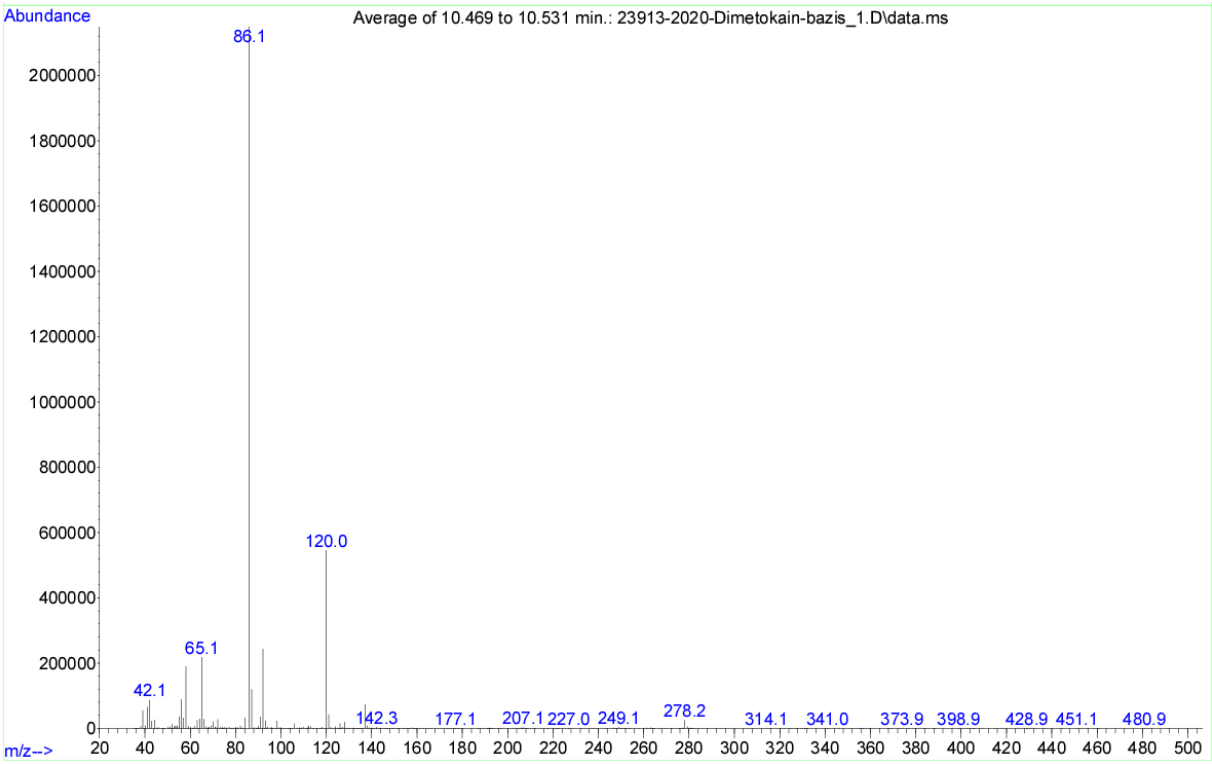
An Agilent 6890N Network GC system set up with Agilent HP-5MS (length: 30 m, diameter: 0.25 mm, film: 0.25 mm) coupled to an Agilent 5973 Network Mass Selective Detector (scan range m/z 35 – m/z 500) was used. The evidence was solved in methanol, the solution was injected. Samples were subjected to electron ionization (EI) mode. GC-MS conditions: HP-5MS column was temperature programmed from 100 ℃ (which was held for 2 minutes) to 280 ℃ at 20 ℃/min, 280°C was held for 3 minutes, then to 315 ℃ at 25 ℃/min, the temperature was stated at 315 ℃ for 12 minutes. The carrier gas was helium. Tribenzyl-amine was applied as an internal standard (locked to 10.8 minutes). Data handling was carried out with GC/MSD ChemStation software.

GC-MS total ion chromatogram of the evidence

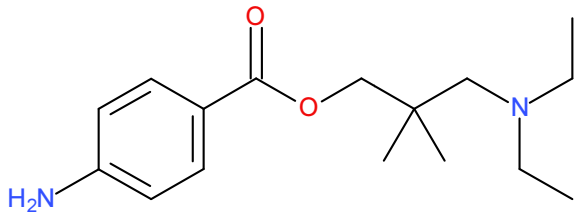


Agilent 6890N Network GC system set up with Agilent HP-5MS

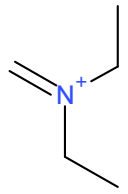
Mass spectrum at 10.50 min retention time



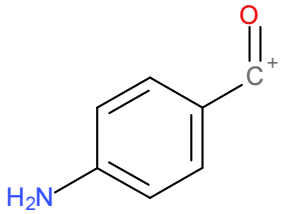
Interpretation of the mass spectrum



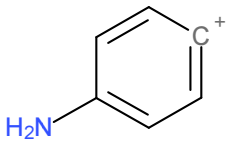
M/z = 278



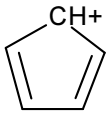
m/z = 86



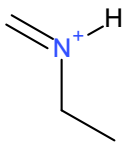
m/z = 120



M/z = 92



m/z = 65



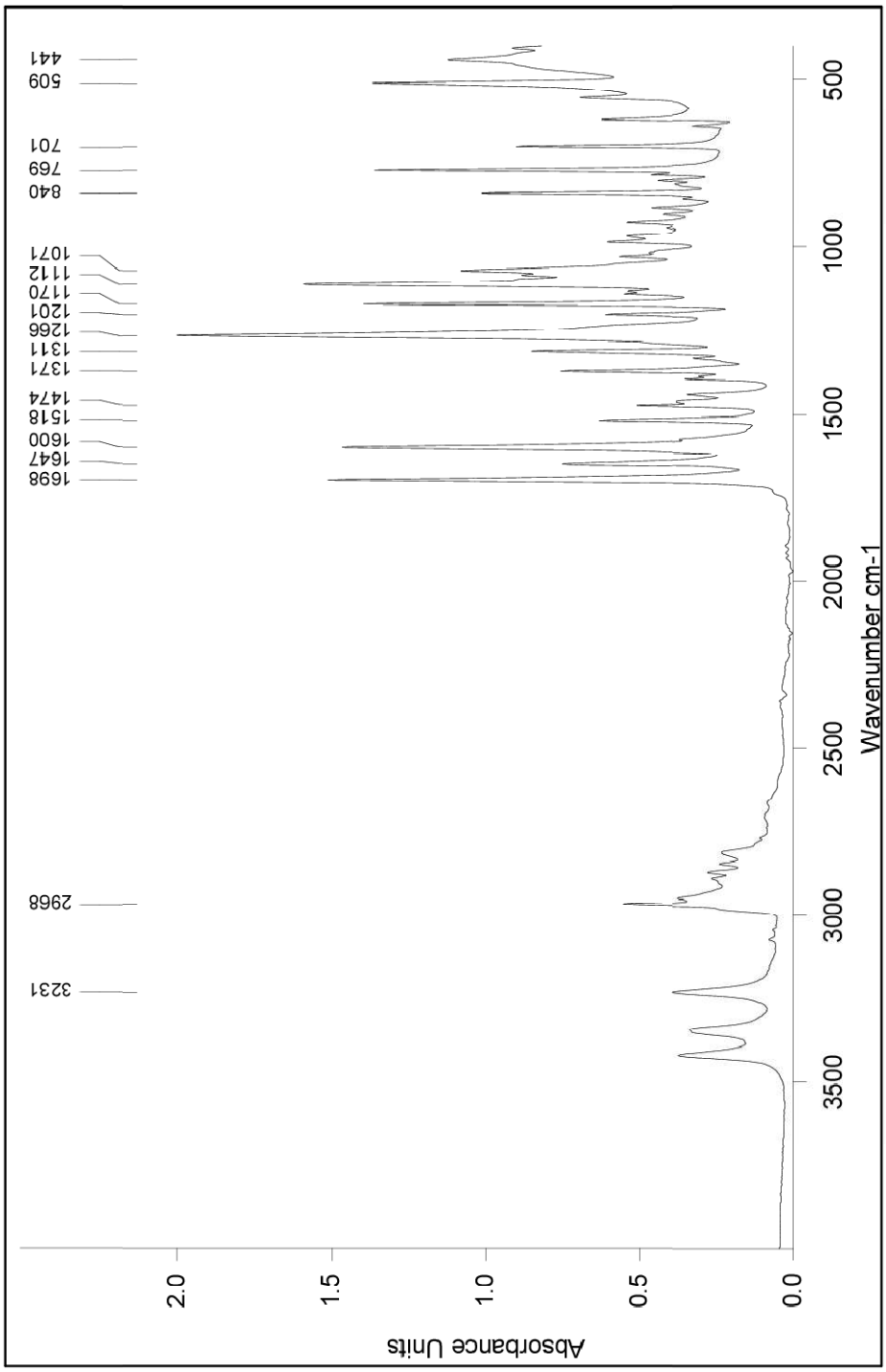
m/z = 58

Agilent 6890N Network GC system set up with Agilent HP-5MS

IR

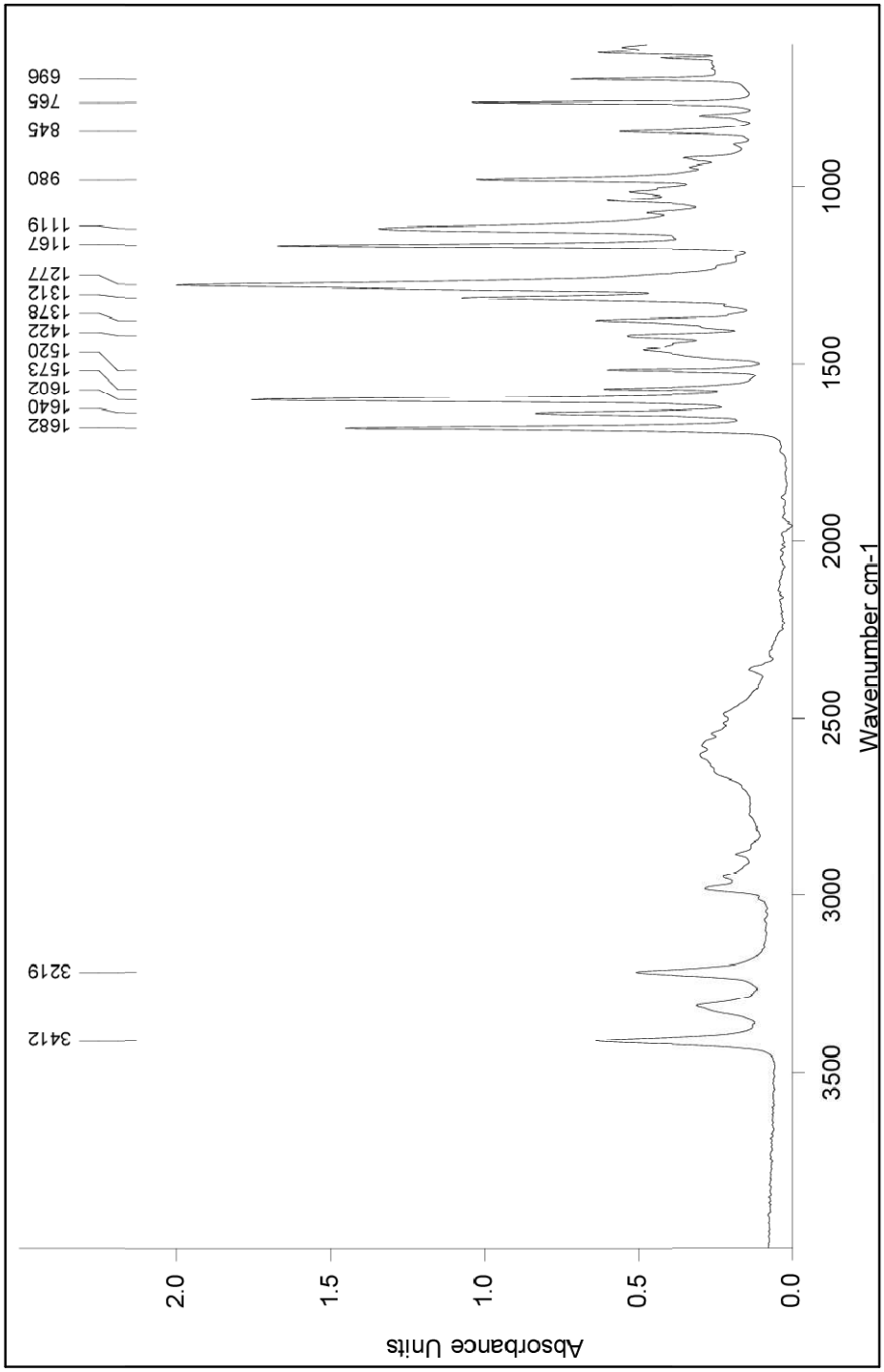
The IR spectrum was recorded on a Bruker Tensor 27 IR spectrometer equipped with a Platinum ATR accessory, in absorbance mode. The digital resolution is 4 cm^{-1}. The milled powder of the sample was measured directly. The spectrometer was controlled, and the data were processed using Opus 6.5 software package.

IR spectrum of the evidence as received



Bruker Tensor 27

IR spectrum of the available reference standard (HCl salt)



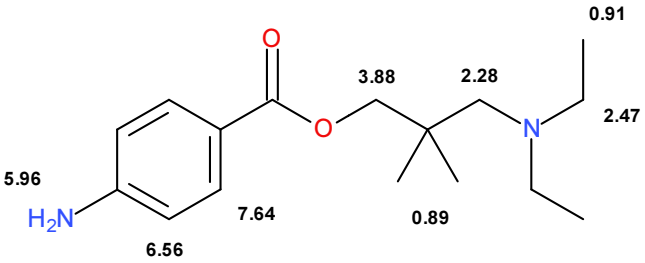
Bruker Tensor 27

NMR

The NMR spectra were recorded on a Bruker Avance Neo 400 NMR spectrometer operating at 9.4 Tesla magnetic field, equipped with Prodigy BBO-H&F-D-05 Z-gradient probe. The spectra were recorded at 25 °C in DMSO-d_6 solution. The spectrometer was controlled, and the data were processed using TopSpin 4.0 software package. Chemical shifts (δ) are given in parts per million unit, referenced to tetramethylsilane (δ_{TMS} = 0.00 ppm). The determination of the structure was based on ^{1}H, zqs-NOESY, as well as ^{13}C, multiplicity edited HSQC, and HMBC spectra.

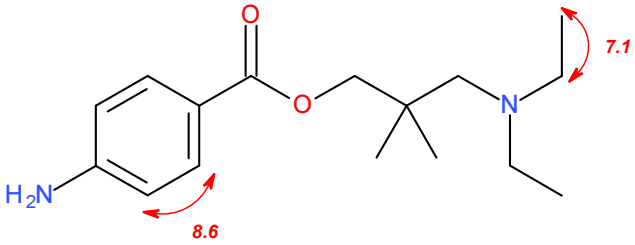
Interpretation of the NMR spectra

^{1}H-NMR chemical shifts δ [ppm]

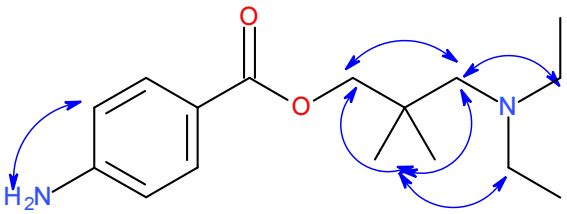


In DMSO-d_6 solution

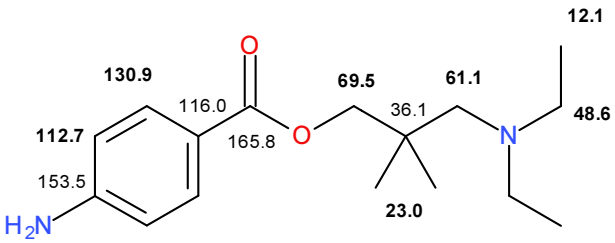
Characteristic J(H,H) coupling constants [Hz]



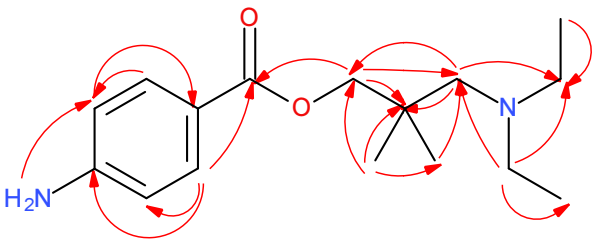
Characteristic H-H steric proximities detected by zqs-NOESY experiment



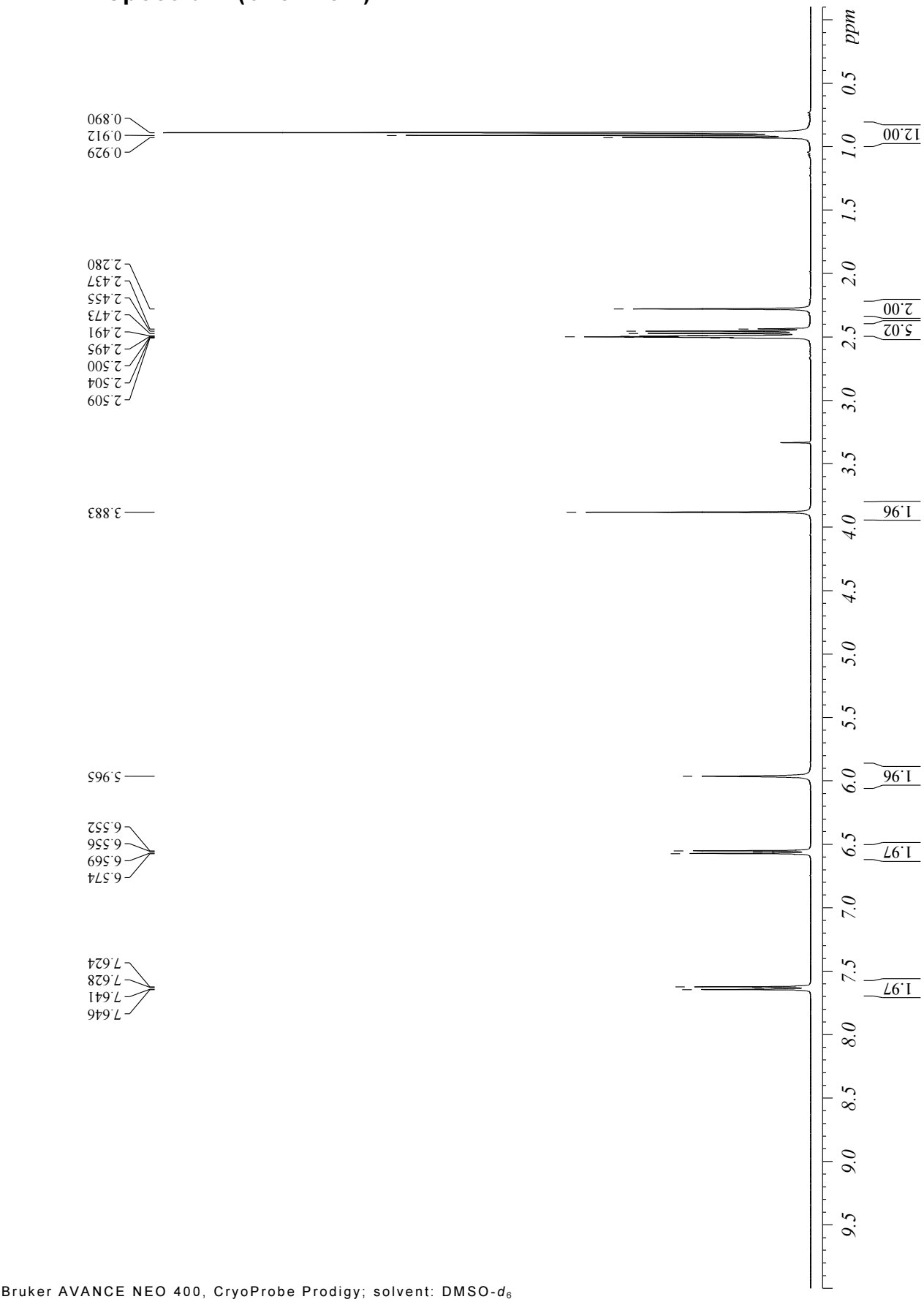
^{13}C-NMR chemical shifts δ [ppm]



Characteristic hteronuclear long-range coupling detected by HMBC experiment
H ⟶ C

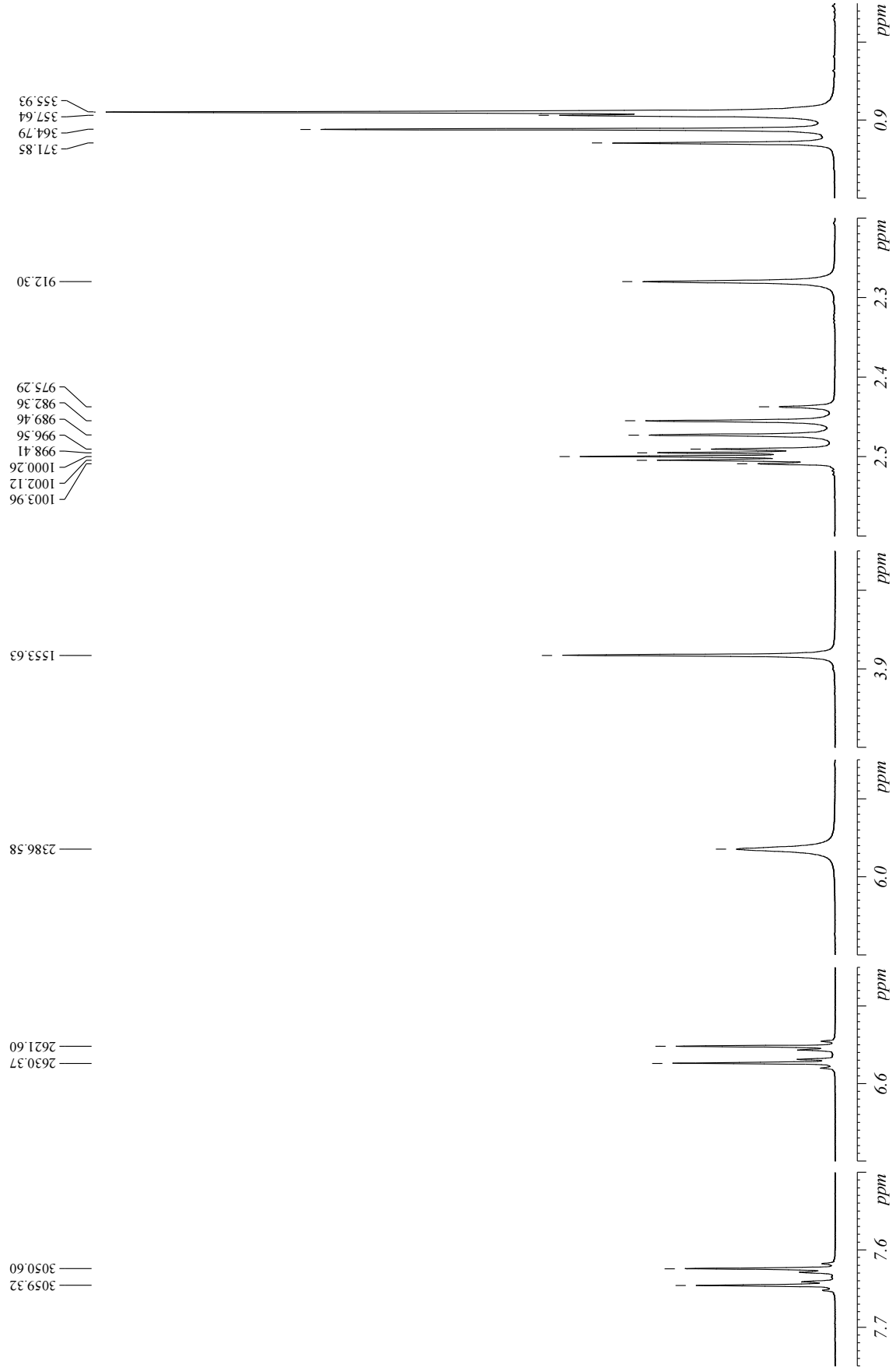


^{1}H-NMR spectrum (overview)



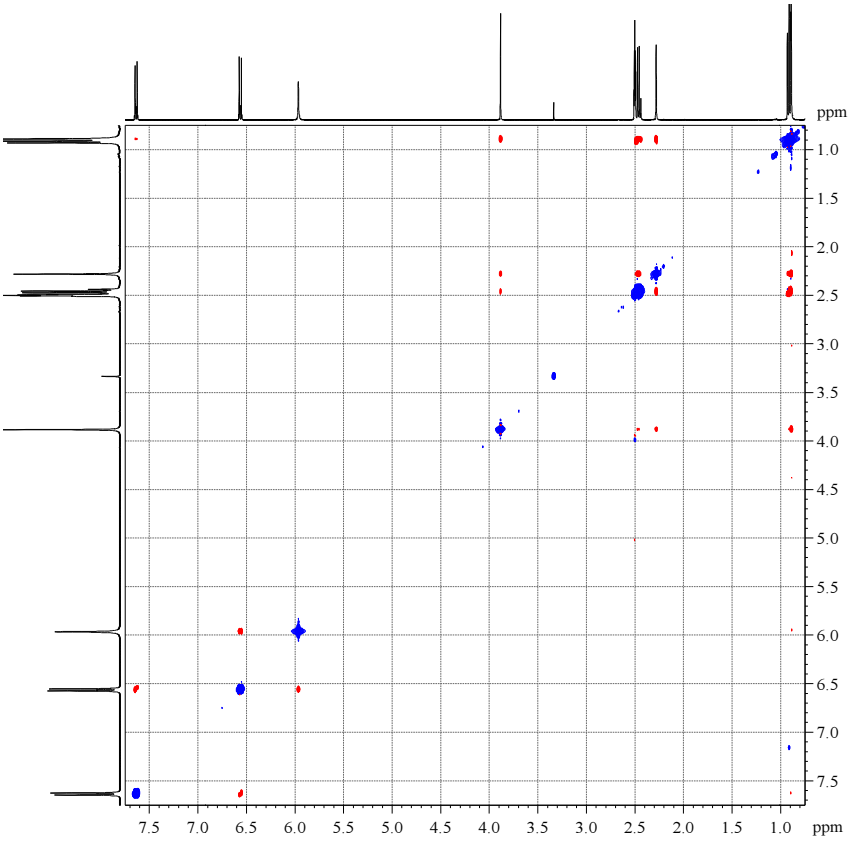
Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

Characteristic sections of the ^{1}H-NMR spectrum

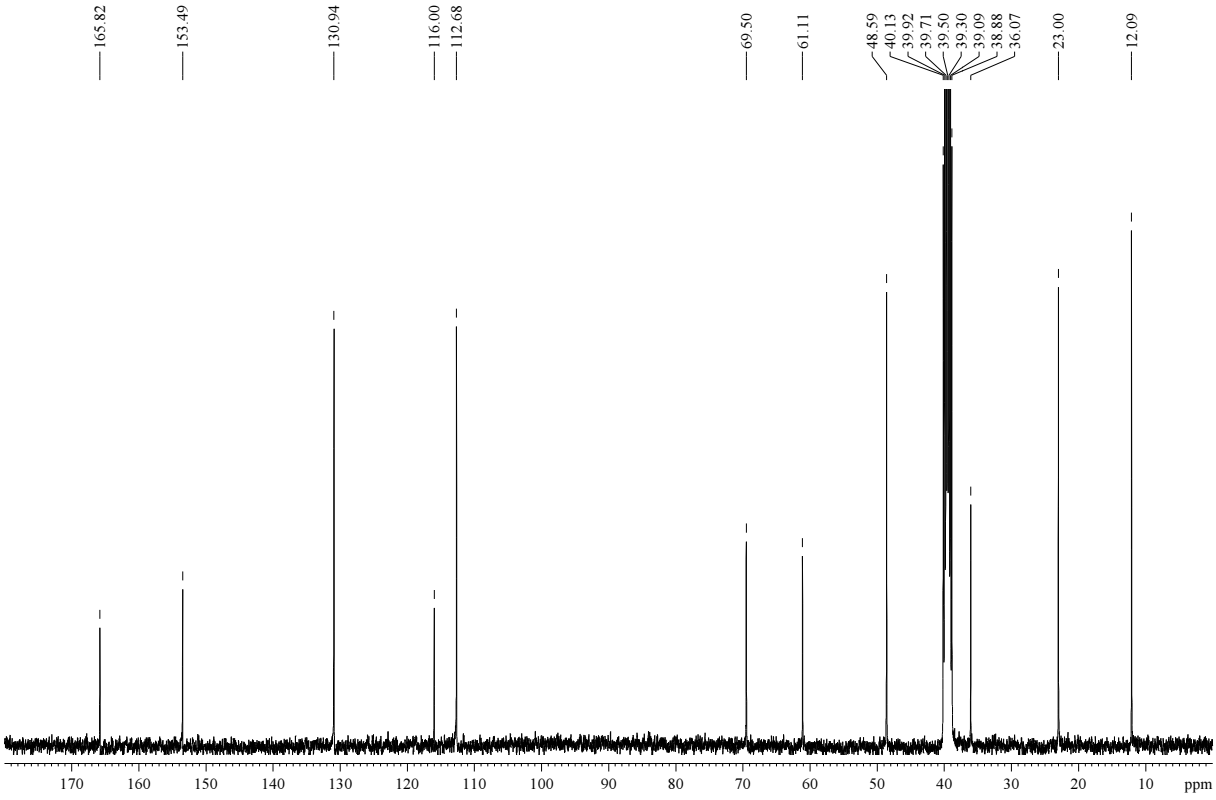


Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

zqs-NOESY

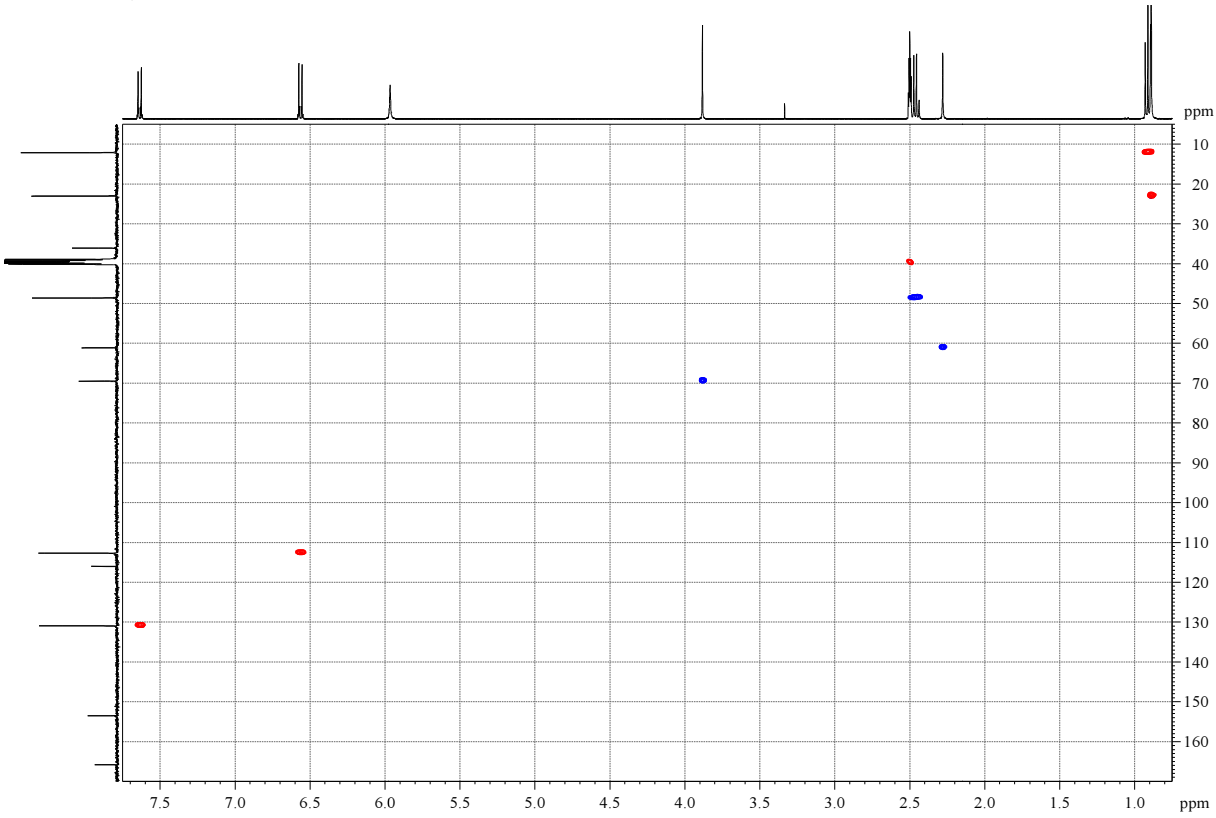


^{13}C-NMR spectrum

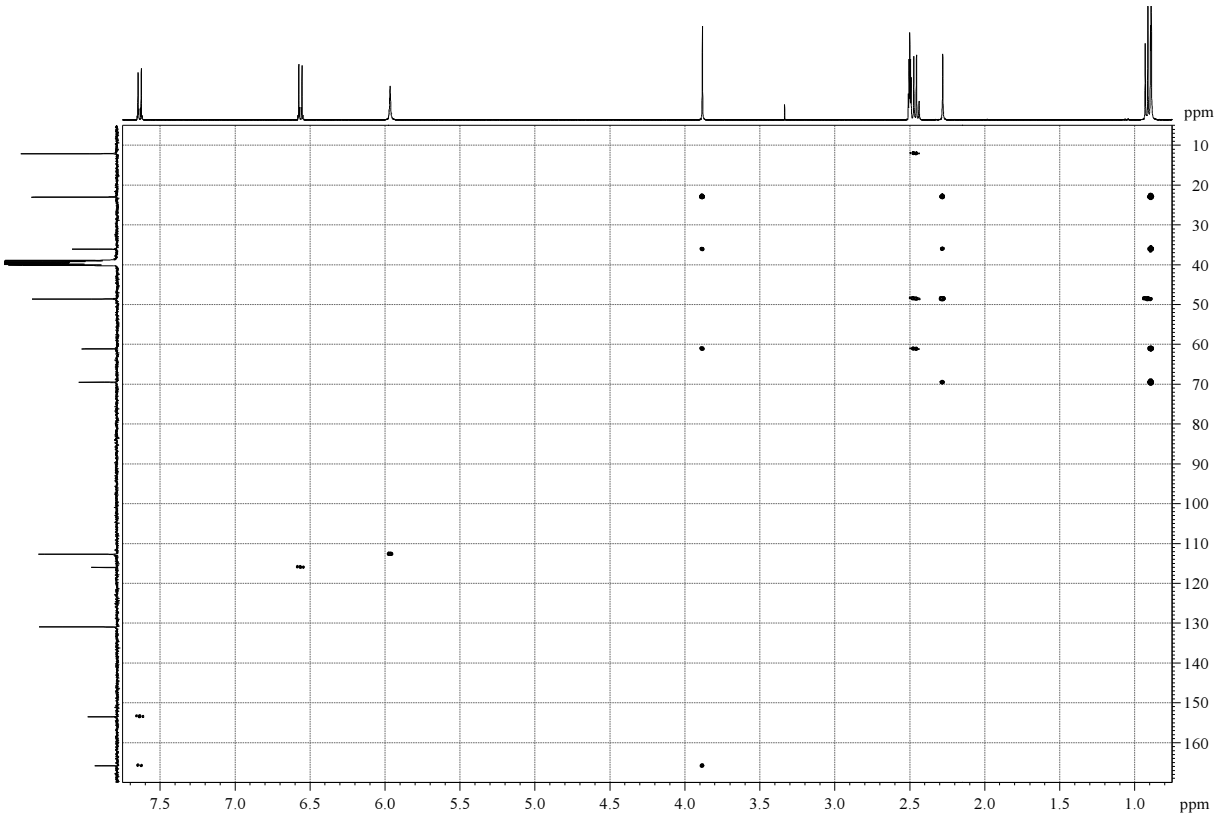


Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

ed-HSQC



HMBC

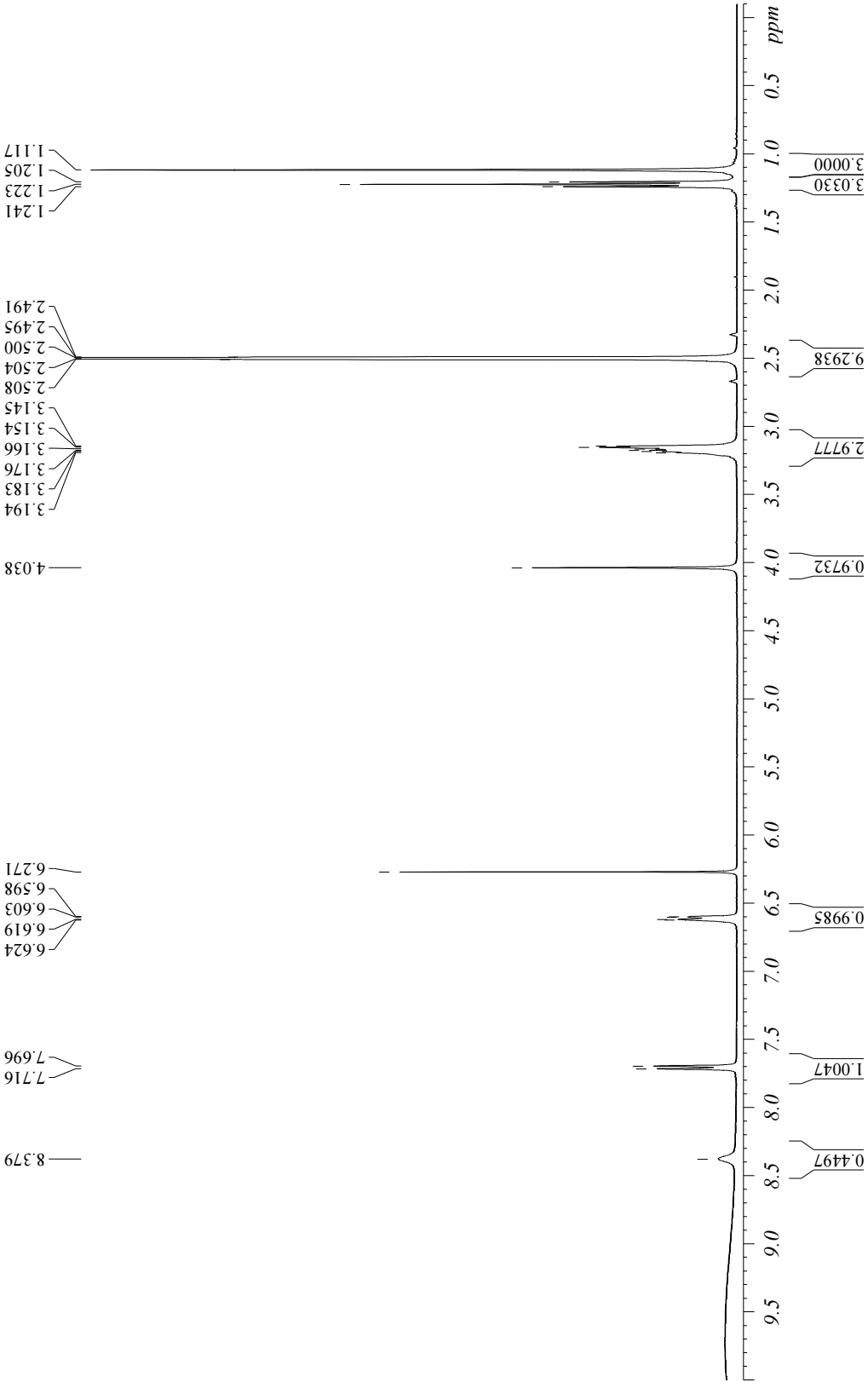


According to the result of the IR and NMR spectra, the seized material is free base.

Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

^{1}H-NMR spectrum of the trifluoroacetate salt of dimethocaine formed *in situ* in the NMR tube.

The solution contains maleic acid internal standard for *q*NMR measurement (signal at 6.27 ppm)



Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

References:

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https://www.caymanchem.com/product/11159/dimethocaine-(hydrochloride)
https://www.caymanchem.com/gcms/11159-0452396-GCMS.pdf

M. R. Meyer, C. Lindaue, J. Welter, H. H. Maurer: Dimethocaine, a synthetic cocaine analogue: studies on its in-vivo metabolism and its detectability in urine by means of a rat model and liquid chromatography-linear ion-trap (high-resolution) mass spectrometry
Analytical and Bioanalytical Chemistry, **406** (7), 1845–1854 (2014).
doi:10.1007/s00216-013-7539-0.

M. R. Meyer, C. Lindauer, H. H. Maurer: Dimethocaine, a synthetic cocaine derivative: studies on its in vitro metabolism catalyzed by P450s and NAT2
Toxicology Letters, **225** (1), 139–146 (2014) doi:10.1016/j.toxlet.2013.11.033